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Improved thermoelectric performance of a film device induced by densely columnar Cu electrode

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ABSTRACT

In this study, it was found that the columnar Cu film is similar as a parallel microchannel which can create some sort of channels for the easy transport of electrons and phonons in the device. The *p*-$Bi_{0.5}Sb_{1.5}Te_3$, *n*-$Bi_2Se_{0.3}Te_{2.7}$ and Cu films were fabricated by a magnetron sputtering method. These films have been integrated into low-dimension cross-plane devices using mask-assisted deposition technology. The performance of the micro-device with densely columnar Cu film electrode has been tested, which was very superior to that of the device with ordinary structure electrode. For the typical device with 98 pairs of *p*/*n* couples, the output voltage and maximum power were up to 120.5 mV and 145.2 μW, respectively, for a temperature difference of 4 K. The device could produce a 14.6 K maximum temperature difference at current of 160 mA. The response time to reach the steady condition was less than 2 S. The results prove that excellent performance of micro-device can be realized by integrating the densely columnar Cu electrode.

1. Introduction

Solid-state thermoelectric (TE) micro-devices have been frequently studied in recent [1–4]. Unfortunately, many non-idealities become apparent and must be considered when moving from bulk to thin-film devices. Whereas electrical and thermal contact resistance and heat generation in the current carrying connections all become critical for thin-film devices [5]. For practically achievable values of modern Bi_2Te_3 heat exchangers, the impact of modern parasitic resistances results in a 50% reduction in the figure of merit at length scales less than ~0.5 μm [6]. Some of the highest performing thin-film TE materials with material *ZT* values in excess of 2 only achieve device performance equivalent to a material *ZT* of less than 0.4 when all the passive losses inherent to the device design are taken into account [7,8], i.e. an effective device $ZT < 0.4$. Joule heating at the metal-semiconductor interface has been a primary component in the reduction of theoretical cooling predictions by as much as 97% [5,9]. The contact resistance for thin-film device is a bottleneck, which badly confines the performance of TE micro-devices. To overcome this issue, numerous research efforts to explore various electrodes for devices have been done [10–14]. These reported methods have some other advantages, for example, excellent electrical conductivity and multilayered structure design for electrodes, but with the drawback of poor electrical and thermal conductivity at the metal-semiconductor interfaces. However, the columnar structuring process possibly induces a favorable change in the Fermi surface topology to improve the problem. Besides, the columnar structure electrode is similar as a parallel microchannel which can create some sort of channels for the easy transport of electrons and phonons in the device.

Among the metal electrodes, Cu is an attractive material, one of the cheapest common metals and an environment friendly product. To improve the properties of Cu as electrode materials for TE micro-devices, an efficient way is to control the structure of Cu nanomaterials by a simple magnetron sputtering method. Using densely columnar film as electrode is one of the most effective approaches to enhance micro-devices performance. Here, it is found that densely columnar Cu film is introduced into a cross-plane device as electrode (reducing Joule heating and improving thermal transport) using magnetron sputtering, which can greatly improve electrical and thermal transport and dramatically enhance performance of a vertical-type micro-device with Bi_2T_3-based thin-film couples. It is also the main emphasis on correlating device performance with electrode structure. Such correlation provides valuable information for the construction of various TE devices and

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provides guidance for thermal management applications requiring simultaneous control over electrical and thermal conductivities.

2. Experimental section

In this work, p-$Bi_{0.5}Sb_{1.5}Te_3$ and n-$Bi_2Se_{0.3}Te_{2.7}$ films were grown at 300 °C deposition temperature and 2 Pa working pressure in a magnetron sputtering system. Commercial 60-mm-diameter hot-pressed $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ and Te (99.99% purity) targets (Purchased from General Research Institute for Nonferrous Metals, China) were used for co-sputtering to compensate for evaporated tellurium at high temperature. The target direct-current powers were set to 35 W and 40 W for $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$, respectively. While the Te target was connected to a radiofrequency power supply with power of 30 W. Cu target (99.99% purity) was sputtered by target power of 25 W for the columnar or the ordinary Cu films at 300 °C deposition temperature and 2 Pa or 1 Pa working pressure, respectively. The base pressure was lower than 2×10^{-4} Pa. Before deposition, AlN substrates were cleaned thoroughly by diluted nitric acid and acetone, and dried under the nitrogen airflow.

The stainless steel masks with designed patterns were used to fabricate device connected electrically in series, and the masks include mask for TE film ($mask_f$) and masks for electrodes ($mask_e$), respectively. Connection metal pads of Cu were first deposited on the lower and the upper AlN plates with thickness of 0.25 mm using magnetron sputtering and $mask_e$-assisted deposition technology, respectively. The size of lower (upper) AlN plate is 35 (30) mm × 30 mm. Then p-$Bi_{0.5}Sb_{1.5}Te_3$ and n-$Bi_2Se_{0.3}Te_{2.7}$ film couples with 1 mm × 1 mm in area were deposited onto metal pads under $mask_f$, respectively. Finally, the lower AlN plate with 98 p-type elements and the upper AlN plate with 98 n-type elements were bonded using flip-chip bonding techniques to form a 98 pairs of p/n couples module. The thickness of TE films is about 2 μm and the Cu electrode thickness is about 500 nm by controlling deposition rate and sputtering time. Before operation, the devices were annealed in N_2 gas at 150 °C for 0.5 h. Annealing improved the mechanical and electrical contacts between the thermoelectric elements and interconnects, and also improved the thermoelectric figure-of-merit of the thermoelectric materials.

The crystal structures of $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ films grown on SiO_2 substrates were examined by X-ray diffraction (XRD, Rigaku D/MAX 2200) using Cu K_α radiation ($\lambda = 0.154056$ nm). The films and couples morphology were observed by field-emission scanning electron microscopy (FE-SEM, Sirion 200). The compositions were detected by energy dispersive X-ray spectroscopy (EDX). Surface profilometry (Ambios XP-2, USA) was used to measure the film thickness. The electrical conductivity (σ) and Seebeck coefficient (S) were simultaneously measured on thin films deposited on $5 \times 15 \times 1$ mm^3 substrates using a ZEM-3 (Ulvac Riko, Inc.). The thermal conductivity (κ) data were collected using a Laser PIT (Ulvac Riko, Inc.) at room temperature. The principle of the measurement method is described in detail in Ref. [15]. The carrier concentration and mobility were determined using a four-probe measurement based on the Hall effects (ECOPIA HMS-3000) at room temperature. We also measured the overall resistance of the TE devices by a voltammetry method. The output voltages of the devices were measured by a DC digital voltage/current meter (Shanghai SB-2238) while applying a temperature difference between the hot and cold sides of the devices.

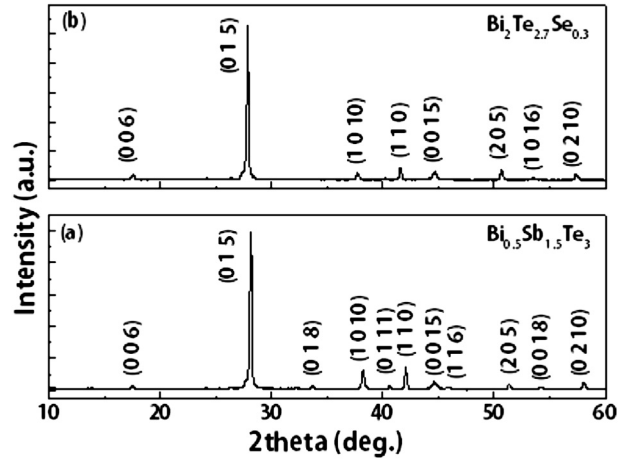

Fig. 1. XRD patterns of (a) $Bi_{0.5}Sb_{1.5}Te_3$ and (b) $Bi_2Se_{0.3}Te_{2.7}$ films.

3. Results and discussion

The p-$Bi_{0.5}Sb_{1.5}Te_3$ and n-$Bi_2Se_{0.3}Te_{2.7}$ films have been synthesized by a simple magnetron sputtering technique at 300 °C deposition temperature and 2 Pa working pressure. XRD patterns of $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ films are shown in Fig. 1. For the both films, all peaks are indexed as rhombohedral phase (JCPDS 49-1713 and 50-0954 corresponding to Fig. 1a and b), implying the formation of polycrystalline structure. The intense and sharp XRD peaks from the films are typical signatures of a high degree of crystallinity. It reveals a single-phase product with slightly broadened reflections, which is typical for crystals with low dimensions.

The columnar and the ordinary Cu films are shown in Fig. 2. Seen from cross-sectional view (Fig. 2a), the columnar film is relatively dense and uniform, and a number of columns are existed in the film. As shown in Fig. 2a, we can observe that a large number of Cu columns are densely grown perpendicular to the substrate. In the film, the diameters of columns are in the range of 40–100 nm. The Cu columns array is similar as a parallel micro channel. By

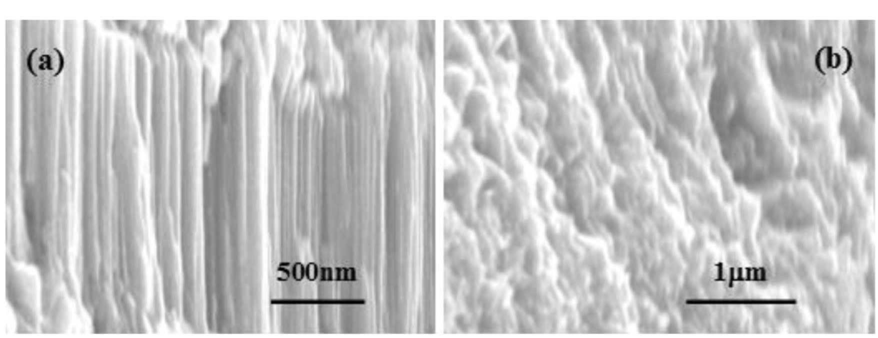

Fig. 2. SEM images of (a) columnar and (b) ordinary Cu films with cross-sectional view.

Table 1
Transport properties of the $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ films measured at room temperature.

Films	Carrier concentration ($10^{19}/cm^3$)	Carrier mobility ($cm^2/V\ s$)	Electrical conductivity (10^4 S/m)	Seebeck coefficient (μV/K)	Thermal conductivity (W/m K)	$ZT \sim 300$ K
$Bi_{0.5}Sb_{1.5}Te_3$	5.1	73	5.9	207	0.96	0.79
$Bi_2Te_{2.7}Se_{0.3}$	−6.8	62	6.5	−196	0.91	0.82

controlling growth parameters, the microstructure of the film obviously changed, as shown in Fig. 2. For working pressure of argon of 1 Pa, the ordinary structure Cu film is obtained (Fig. 2b), which is composed of numerous disordered particles.

Subsequently, the transport properties of the $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ films were studied at room temperature, such as, the concentration and mobility of carriers, the electrical conductivity, the Seebeck coefficient, and the thermal conductivity, as shown in Table 1. In addition, the electrical conductivities of the columnar and the ordinary Cu films investigated by a ZEM-3 show that both films have a similar electrical conductivity of $\sim 2 \times 10^7$ S/m at 300 K. These films may provide excellent properties for fabrication of a device near room temperature.

Thus, the $Bi_{0.5}Sb_{1.5}Te_3$, $Bi_2Se_{0.3}Te_{2.7}$ and Cu films were then introduced into a TE micro-device using mask-assisted deposition technology. Fig. 3 shows photographs of the 98 pairs of p/n couples device. According to a two-wafer concept, each pad is occupied in this stage with only one TE material type. Fig. 3(a) and (b) shows photographs of the p- and n-single chip constituted by $Bi_{0.5}Sb_{1.5}Te_3$ and $Bi_2Se_{0.3}Te_{2.7}$ films couples, respectively. The n-single chip is aligned to the complementary p-side and then bonded together using flip-chip bonding techniques (see Fig. 3c).

Fig. 4a and b displays the cross-section SEM images of the bilayer structure for $Bi_{0.5}Sb_{1.5}Te_3$/Cu and $Bi_2Te_{2.7}Se_{0.3}$/Cu films couples, respectively, corresponding to the device with columnar electrode. It is observed that $Bi_{0.5}Sb_{1.5}Te_3$, $Bi_2Se_{0.3}Te_{2.7}$ and columnar Cu structure have been successfully incorporated into the micro-device. It exhibits detailed morphology of the Cu electrode/TE Bi_2Te_3-based film interconnects applied to ensure good electrical contact, which is the key to guaranteeing good device performance. For comparison, the device with ordinary Cu film electrode was also fabricated. The EDX results confirm that the atomic ratios are quite similar to those of the targets in these films couples.

Next, the performance of film devices was measured. The internal resistances (R_{in}) of the devices were obtained by a voltammetry method. The R_{in} value of the device with columnar Cu electrode is 25 Ω (Table 2), which is much smaller than that of the device with ordinary electrode (56 Ω). The resistance (R_f) of the film materials in the devices is calculated according to the similar method described in Ref. [16]. The R_f value is about 21 Ω owing to high electrical conductivity of film materials in both devices. The electrical contact resistance ($R_c = R_{in} - R_f$) of the interconnection between the electrodes and TE films is about 4 Ω or 35 Ω for the device with columnar or ordinary Cu electrode, respectively. This implies that the densely columnar Cu electrode can significantly improve the electrical contact resistance.

To measure the open output voltage under a temperature difference for the device, one side of the TE element was heated and the other side was cooled using a self-made adapted characterization appliance. The temperature gradient was imposed parallel to the length of the TE legs. The output voltages of the device were obtained by a DC digital voltage meter. The maximum output power was estimated from the open output voltage and internal resistance of the device.

Fig. 5 shows the variation of the open output voltage (V_{op}) and output power (P_{max}) with temperature difference applied across

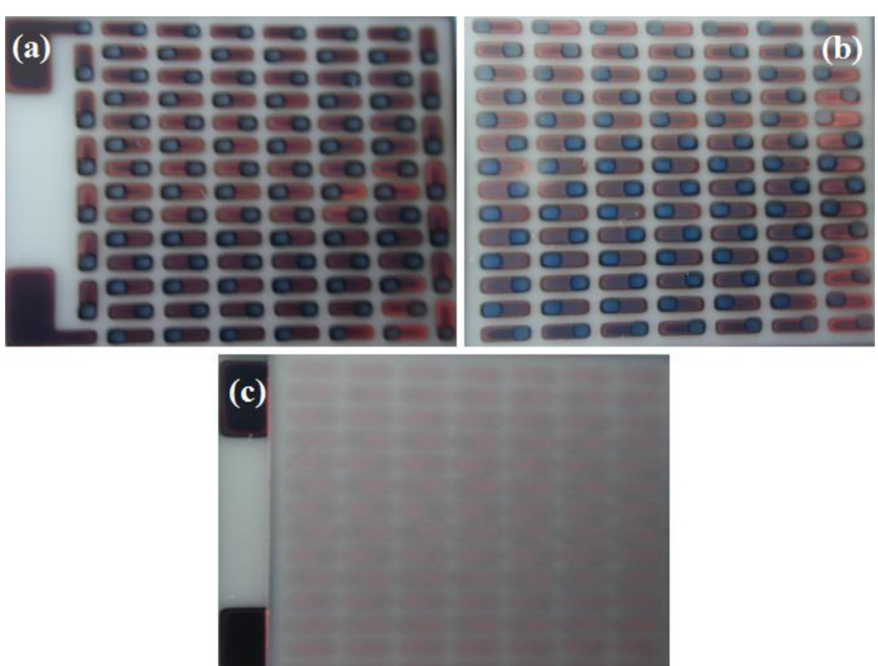

Fig. 3. Photographs of (a) lower AlN plate with 98 p-$Bi_{0.5}Sb_{1.5}Te_3$ elements, (b) upper AlN plate with 98 n-$Bi_2Se_{0.3}Te_{2.7}$ elements, (c) 98 pairs of p/n couples device.

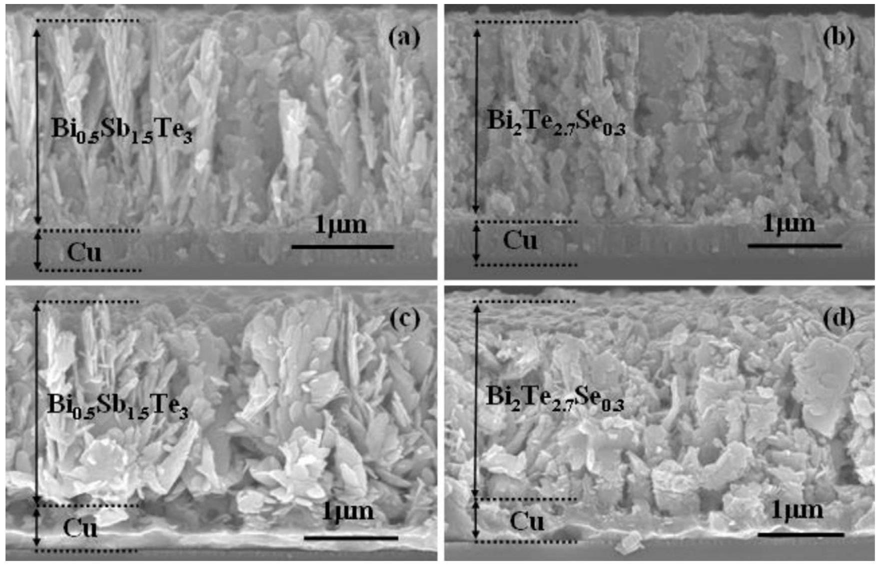

Fig. 4. Cross-section SEM images of the couples in devices with (a,b) columnar and (c,d) ordinary Cu film electrodes.

Table 2
The highest open output voltage, output power and cooling performance of devices.

Devices (electrode)	R_{in} (Ω)	R_c (Ω)	Power-generator			Cooler	
			ΔT (K)	V_{op} (mV)	P_{max} (μW)	I (mA)	ΔT_{max} (K)
Columnar	25	4	4	120.5	145.2	160	14.6
Ordinary	56	35	4	64.6	18.6	160	7.2

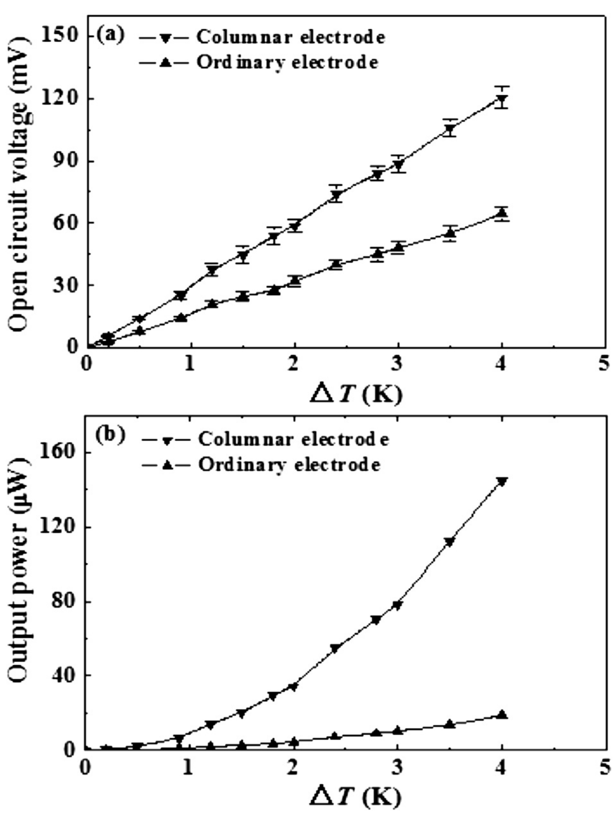

Fig. 5. (a) Open output voltage and (b) output power for devices with columnar and ordinary electrodes as a function of applied temperature difference (ΔT).

the device. As seen from Fig. 5(a), V_{op} approximately linearly increases with the temperature difference, which also implies that S remains constant with test temperature. Unlike for bulk Bi_2Te_3-based materials, whose Seebeck coefficients decrease as the temperature increasing. The temperature stability of S of the Bi_2Te_3-based films is useful when the TE device is in practical use. Compared with the ordinary device, the V_{op} and P_{max} values of the device with columnar film electrode are greatly enhanced.

The highest values of V_{op} and P_{max} obtained for the device with columnar film electrode used as a power generator are 120.5 mV and 145.2 μW at $\Delta T = 4$ K, respectively (Table 2). P_{max} of the device with columnar film electrode reaches 7.8 times larger than that of the device with ordinary film electrode at $\Delta T = 4$ K. This implies that the electrical contact resistance decreases due to using columnar structure electrode, thus greatly enhancing the performance of TE micro-device. For comparison, some devices integrating bismuth-antimony-telluride-based TE films, such as the TE generator developed by Francioso et al. [16] with 100 thermocouples of n-Bi_2Te_3 and p-Sb_2Te_3 [each leg with 2 mm (length) × 250 (425) μm (width) × 0.5 μm (thickness)], could produce a maximum open-circuit voltage of 430 mV and P_{max} of 32 nW at $\Delta T = 40$ K; Takashiri et al. [17] reported a highest output voltage of 83.3 mV and estimated output power of 0.21 μW from a TE generator containing seven pairs of p-type $Bi_{0.4}Sb_{1.6}Te_3$ and n-type $Bi_2Te_{2.7}Se_{0.3}$ thin films [each leg with 15 mm (length) × 1 mm (width) × 1 μm (thickness)] at $\Delta T = 30$ K; Kwon et al. [18] fabricated a generator based on 20 pairs of p-type $Bi_{0.4}Sb_{1.6}Te_3$ and n-type Bi_2Te_3 films, each leg with 12 mm (length) × 200 μm (width) × 4.0 μm (thickness), which could only achieve $P_{max} = 1.3$ μW at $\Delta T = 45$ K; Shin et al. [19] prepared a micro-TE thin-film generator (device area 12 × 12 mm^2) with 11 couples of p-type BiSbTe and n-type Pt, which could generate 1.9 μW of power at $\Delta T = 50.6$ K; Kim et al. [20] fabricated a device with 242 pairs of n-Bi_2Te_3 and p-Sb_2Te_3 film legs, each leg with 100 μm (diameter) × 20 μm (thickness), which could achieve a maximum open-circuit voltage of 294 mV and $P_{max} = 5.9$ μW at $\Delta T = 22.3$ K, the presented device with columnar film electrode shows much better performance. However, these reported devices have some other advantages, for example, excellent V_{op} and good design concept, but with the drawback of high R_{in} due to the large resistance of materials and parasitic resistances. However, the presented film device with columnar film electrode possesses relatively low R_{in}, leading to improved P_{max}. The

columnar Cu film electrode is similar as a parallel microchannel which can create some sort of channels for the easy transport of electrons and phonons in the device (reducing Joule heating and improving thermal transport). However, the measured output voltages of the devices are lower than the predicted results based on the Seebeck coefficients values of 207 μV/K and −196 μV/K for the TE films. This may result from a structural anisotropy of films, thermal contact effect, heat loss problem [21], or electric losses at the interconnects because of the contact resistances.

Based on the Peltier effect, the holes or electrons in the $Bi_{0.5}Sb_{1.5}Te_3$/$Bi_2Se_{0.3}Te_{2.7}$ junction will absorb sufficient energy to be transported to a higher energy level as the driving current flows from one side of the *p*-$Bi_{0.5}Sb_{1.5}Te_3$ or *n*-$Bi_2Se_{0.3}Te_{2.7}$ leg toward the other side. Therefore, this junction will serve as a heat absorber. Fig. 6 shows the sub-ambient cooling obtained in the device with columnar Cu film electrode ($T_{C,H}$ or $T_{C,C}$ is the heat or cold side temperature of the device with columnar electrode, respectively.). With an ambient temperature T_A of about 15.5 °C, we have 8.5 K of cooling (down to 7 °C) and 14.6 K maximum temperature difference at current of 160 mA without any forced heat removal by blowing air or running water at the heat sink. Under similar testing conditions, we measure 3.9 K of cooling and 7.2 K maximum temperature difference in the device with ordinary Cu film electrode, as shown in Fig. 6 ($T_{O,H}$ or $T_{O,C}$ is the heat or cold side temperature of the device with ordinary electrode, respectively.). The cooling performance of the micro-device with densely columnar Cu film electrode is very superior to that of the device with ordinary electrode. The columnar Cu film is introduced into the device, which has greatly improved electrical and thermal transport and dramatically enhanced performance of a TE micro-device. The results prove that excellent performance of micro devices can be realized by integrating the densely columnar Cu electrode. Besides, the response time [22,23] to reach the steady condition is less than 2 S in the device with columnar Cu film electrode, while the response time is about 3 S for the device with ordinary electrode. This difference of response times is supposed to be mainly related to the electrodes of devices. The heat transfer for columnar structure electrode is faster than that of the ordinary structure electrode, which can be understood by the thermal response time [24]. The thermal response time is about $4l^2/\pi^2 D$, where l is the thickness of film and D is the thermal diffusivity. In the ordinary film electrode, it lengthens electron/phonon transport journey and decreases thermal diffusivity along cross-plane direction due to multiple scattering, that is, l becomes relatively large and D becomes small in fact. While the columnar film electrode is similar as a parallel microchannel which can create some sort of channels for the easy transport of electrons and phonons, indicating that l is smaller and D is larger in comparison with the ordinary structure electrode. One-dimensional columnar structure is a short possible way or dimension for the flow of electrons and phonons. This faster response for columnar Cu film electrode is demonstrated by the above analysis, i.e., that's why faster heat transfer for the device with columnar Cu film electrode in comparison to the device with ordinary structure electrode.

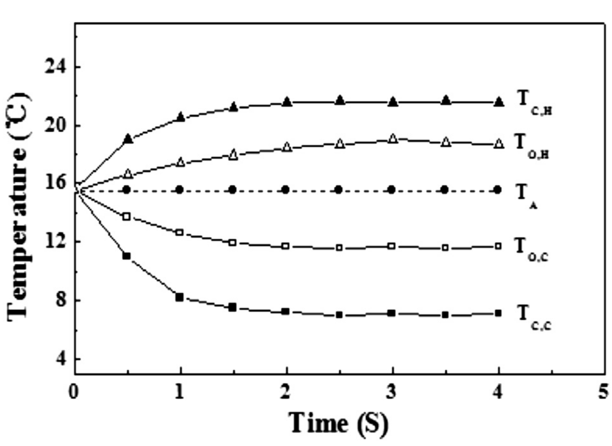

Fig. 6. Temperature for the hot/cold-sides of devices as a function of time at 160 mA current.

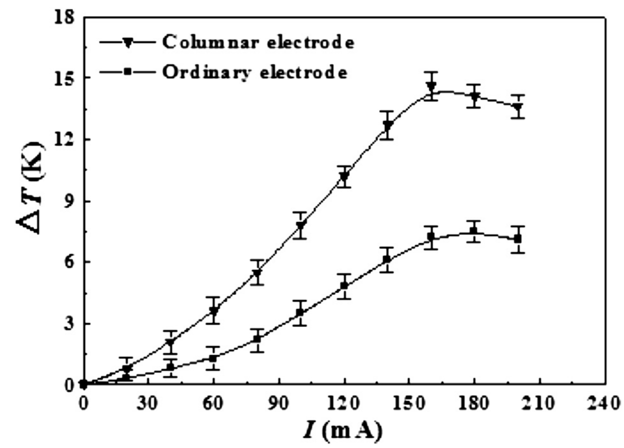

Fig. 7. Temperature difference (ΔT) versus input current for the devices with columnar and ordinary electrodes.

Cooling of the devices was further examined, and all tests were repeated 10 times, as shown in Fig. 7. It is noticed that ΔT increases with increasing current to a certain optimum value of current, then slowly decreases. At room temperature, the device with columnar electrode can produce ΔT_{max} = 14.6 K at current of 160 mA. Compared to the device with ordinary electrode, it suggests that good cooling by a TE device is associated with high $S^2\sigma$ and small R_{in} (Fig. 7; Table 2). This further confirms that the columnar structure electrode is beneficial for improving the performance of devices. In comparison with previous research (see Table 3), it is noted that the previously reported devices possess low TE film thermopower or high device electrical resistance, which degrade the cooling produced in these devices. These adverse factors are improved in our device, enabling the device to show relatively good cooling performance. However, the results are far from the predicted results due to the relatively high electrical contact resistances between the metallic junctions and TE elements, and radiation losses, and the thermal conductance of the substrate, the cooling power loss from the side contact, etc. It is expected that these electrical contact resistances can be further reduced by optimizing Cu electrode structure. The optimized aspect ratio of the thermoelements also remains to be explored.

4. Conclusion

The 98 pairs of *p*-$Bi_{0.5}Sb_{1.5}Te_3$/*n*-$Bi_2Se_{0.3}Te_{2.7}$ couples film device with densely columnar Cu electrode was successfully fabricated using the magnetron sputtering and mask-assisted deposition

Table 3
The cooling performance of as a function of currents applied to the devices.

Devices	$S^2\sigma$ (mW/m K^2)	R_{in} (Ω)	I (mA)	ΔT (K)	References
98 Pairs of *n*/*p*	2.5/2.53	25	160	14.6	Our work
200 Pairs of *n*/*p*	0.3/0.08	/	200	1.2	[25]
60 Pairs of *n*/*p*	0.15/0.3	51	23	1.3	[26]
126 pairs of *n*/*p*	/	/	110	2	[27]

technology. The densely columnar Cu film was introduced into the device as electrode, which has greatly improved electrical and thermal transport and dramatically enhanced performance of the TE micro-device. The columnar Cu film is similar as a parallel microchannel which can create some sort of channels for the easy transport of electrons and phonons in the device. For the typical device with 98 pairs of p/n couples, the output voltage and maximum power were up to 120.5 mV and 145.2 μW, respectively, for a temperature difference of 4 K. The device could produce a 14.6 K maximum temperature difference at current of 160 mA. The response time to reach the steady condition was less than 2 S. Introduction of such densely columnar Cu architecture into micro-devices as electrode is therefore a very promising approach.

Acknowledgments

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